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Thermomechanical and structural analysis of green hybrid composites based on polylactic acid/biochar/treated *W. filifera* palm fibers

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A R T I C L E I N F O

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A B S T R A C T

The purpose of this study is to investigate the chemical treatment impact on *Washingtonia filifera* (WF) fibers using sodium bicarbonate (10% $NaHCO_3$) for varying durations (24, 48, 72, 120, and 168 h) on the physical and mechanical characteristics of polylactic acid (PLA)/WF-biochar/biomass hybrid biocomposites. Differential scanning calorimetry, thermogravimetric analysis, and Fourier transform infrared spectroscopy were employed to examine the temperature effect on the mineralogical composition of treated and raw WF fibers and monitor their thermal behavior. The produced biocomposites' tensile, impact, flexural, and morphological characteristics were evaluated. The results demonstrate that chemical treatments improve matrix-fiber adhesion and remove impurities from the fiber surface. Hybrid biocomposites of PLA biopolymers manufactured from biochar and WF fibers given a sodium bicarbonate treatment for 72 h exhibit improved mechanical properties, such as elasticity modulus and strength under tensile and flexural forces. Consequently, these new hybrid biocomposites can be used in various structural and non-structural products, such as car interiors, new 3D printer filament, biomedical equipment and materials, sports equipment, food packaging.

1. Introduction

Polylactic acid (PLA) biocomposites based on plant fibers are a type of composite material that combines PLA, a biodegradable and renewable polymer, with natural fibers to improve its mechanical properties and lessen its influence on the environment [1–6]. These biocomposites can be used in various structural and non-structural commercial products, such as streetcar and train interiors, automotive parts, biomedical equipment and materials, biodegradable protective [7], sports equipment, electronic components, and food packaging [8]. Combining PLA and natural fibers results in a lightweight material with good mechanical properties and improved durability compared to traditional composites [9]. Several investigations have been dedicated to characterizing treated or untreated plant fibers, particularly jute [10–13], the date palm [14–18], the bamboo [19–21], the coir [22–24], the sisal [25–27], the flax [28–30] and the flower agave [31]. Mudoi and Sinha [32] examined how the bhimal fiber thermally degrades using a non-isothermal thermogravimetric analysis (TGA). The TGA analysis revealed that the highest mass reduction (>60%) was observed within 200–435 °C. This temperature range is commonly utilized for processing specific types of thermoplastics. The research conducted by Devnani and Sinha [33] focused on the extraction, activation energy, characterization, and pyrolysis rate of mêlée fibers (cane grass). A 5% alkaline treatment significantly improved the thermal properties and morphological and mechanical characteristics. Indran and Raj [34] conducted a study to examine the chemical, thermal, anatomical, and mechanical aspects of

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the natural cellulose fiber extracted from the stem of cissus quadrangularis. The TGA results exhibit a thermal stability up to a temperature of 270 °C, corresponding to the temperature during polymerization.

Biochar is a recently developed carbonaceous substance generated from readily available and renewable resources through pyrolysis, typically carried out at moderate temperatures ranging from 350 to 700 °C [35–37]. Biochar is used in various fields, such as soil and water treatment [36], carbon capture [38], and absorption of organic pollutants [39,40]. Biochar has also been used for thermal storage applications [41]. Utilizing biological nanoparticles for exploration unveils promising new perspectives in the field of research. Activated carbon, derived from biochar, stands out because of its chemical stability, widespread availability, high specific surface area, and low density [42]. Atinafu et al. [43] conducted a study where they created a hybrid nanoparticle by combining bamboo biochar and multiwalled carbon nanotubes (MWCNTs) to achieve efficient encapsulation of composite phase change materials (PCM). The bamboo biochar in its original state demonstrated reduced latent heat and PCM loading compared to the MWCNT-bamboo hybrid biochar. This discrepancy was attributed to narrower pores and stronger intermolecular attraction between the functional groups and PCM. In contrast, Alshahrani and Prakash [44] examined how additives derived from high-cellulose corn leaf fibers and sustainable biochar obtained from orange peels affect epoxy-based filament production biocomposites' dynamic and static mechanical properties. Anerao et al. [45] integrated biochar derived from rice husk into PLA to create biocomposite filaments compatible with fused deposition modeling three-dimensional (3D) printing technology. The findings indicated that the inclusion of 5% biochar in PLA resulted in an elastic modulus of 1103 MPa and a maximum tensile strength of 36 MPa. However, flexural strength decreased after the addition of biochar. Manshor et al. [46] studied the thermomechanical and morphological behavior of PLA biocomposites based on durian fibers. The results show that treated durian fibers significantly enhance PLA-based biocomposites' properties and thermal stability compared with untreated biocomposites. Research by Orue et al. [47] focused on how surface modifications affected the properties of sisal fibers and the ability of the fiber/PLA interface to adhere to each other. They showed that alkaline treatment eliminated specific non-cellulosic components (hemicelluloses, lignin). Following treatment, sisal fiber tensile strength dropped while interfacial shear strength values increased by at least 120%.

Jiang et al. [48] have developed biodegradable nanofibrous membranes based on PLA to cope with increasing air pollution and the emergence of epidemic diseases. Liang et al. [49] have made significant advancements by developing self-rechargeable, breathable, and antibacterial air filters using PLA nanofibers through a surface engineering approach involving ultra-small electroactive nano-hybrids. Finocchio et al. [50] investigated the physicochemical and mechanical performances, as well as the water absorption behavior, of biopolymers based on degradable PLA loaded with glass fibers. Flexural and tensile tests revealed material softening and enhancement in sample plasticity as temperature increases. The samples showed signs of embrittlement at high temperatures, suggesting that the biopolymers' degree of crystallinity had increased. Their findings indicated that PLA-based composites are well-suited for applications that do not necessitate prolonged exposure to high temperatures. Huda et al. [51] noted that the thermomechanical behavior of stratified PLA composites based on kenaf fibers depended on how the silane and alkalization treatments modified the Kenaf fibers. Alkali-treated and silane-treated fibers exhibit superior mechanical characteristics compared to biocomposites based on raw fibers. The mechanical characteristics of the biocomposite based on fibers treated first with alkali and then with silane have also been significantly improved.

Rajeshkumar et al. [3] studied the behavior of PLA biocomposites based on natural fibers. They indicated that incorporating natural fibers enhanced the biocomposite's mechanical and thermal characteristics. The PLA matrix's strong interfacial adhesion and physical entanglement are responsible for this improvement; adding natural fibers improves PLA wear performance. Sreekala et al. [52] investigated the impact of chemical treatment on the surface of oil palm fibers. They noticed that applying silane or sodium hydroxide to these fibers raised their elastic modulus. Morrison et al. [53], Jacob et al. [54], Ray et al. [55,56] and Mwaikambo and Ansell [57] also observed similar results on other natural fibers. Kriker et al. [58] indicated that processing date palm fibers (DPFs) in an alkaline environment significantly decreases mechanical characteristics and reduces fiber diameter. For fibers treated with a saturated solution of calcium hydroxide for six months, with diameters of 0.8 and 0.4 mm, they observed that the 0.8 mm diameter fiber retained 69% of the initial tensile strength compared to 10% for the 0.4 mm fiber.

Benzanache et al. [59] utilized a novel composite for plaster-based construction material, integrating fibers extracted from the *Washingtonia filifera* (WF) palm. Their objective was to examine the impact of $NaHCO_3$ processing on the mechanical characteristics of the resultant biocomposite. They found that integrating WF fibers into the gypsum matrix notably boosts this novel material's mechanical strength and ductility. As per the optimization study, the optimal $NaHCO_3$ concentration is 20%, with an ideal treatment duration of 168 h, aligning well with experimental outcomes.

Lekrine et al. [60] investigated the mechanical characteristics of different high-density polyethylene (HDPE) biocomposites fortified with varying proportions of WF fibers (10, 20, and 30% by weight). They provided evidence that the addition of WF fibers to virgin HDPE improves various mechanical properties, including Young's modulus, flexural modulus, flexural strength, and tensile strength. However, a slight reduction in impact resistance was observed when WF fibers were incorporated. Moussaoui et al. [61] examined the impact of physicochemical treatments on the fiber properties of ampelesmos mauritanicus. X-ray diffraction demonstrated that these fibers had a high degree of crystallinity, measuring 52.39%. Through physical processing using 550 W microwaves, the fiber density significantly increased from 1.00 to 1.55 g/cm^3. Moreover, their elastic modulus experienced a notable enhancement, rising from 11 to 18.6 GPa, while their tensile strength surged from 155 to 290 MPa.

Recently, Tablit et al. [62] examined the utilization of arundo (Arundo donax L.) fiber as a reinforcing agent in 3D printing filaments in conjunction with PLA/polypropylene waste. They showed increased Young's modulus and tensile strength while maintaining stability during Izod impact testing. Notably, the alkaline treatment of the arundo fiber significantly reduced the composite's water absorption, with a 64% reduction compared to the untreated fiber-based composite. Ju et al. [63] developed an innovative method for fabricating biocomposites using PLA and lignin modified with polyethylene glycol (PEG), employing a twin-screw extrusion process. They revealed that incorporating PEG-modified lignin improved the heat resistance of the PLA-based biocomposite. The PLA mechanical properties are not affected by containing PEG-modified lignin at concentrations up to 30%. Compared to the PLA-L30 biocomposite, the PLA-PL30 biocomposite demonstrated a 78.9% increase in elongation at break and a 26.4% increase in tensile stress.

For the first time, this study unveiled the impacts of $NaHCO_3$ processing on the thermal characteristics and morphology of WF fibers, which exhibit similarities with other cellulose fibers. Both treated and raw WF fibers, subjected to various treatment durations (24, 48, 72, 120, and 168 h), were meticulously characterized employing a variety of methods, such as scanning electron microscopy (SEM), differential scanning calorimetry (DSC), Fourier transform infrared (FTIR) and TGA. The treated WF fibers and biochar (Bi) have been integrated as reinforcement in PLA-based biocomposites with 23% and 1% mass fractions. The innovative biocomposites were characterized using various techniques, including SEM, tensile, flexural, and Izod impact tests.

2. Experimental methods

2.1. Fibers and hybrid biocomposites preparation

The WF fibers used in this work were collected in the region of Skikda, Algeria, where the geographical coordinates are 36°51′00 'N 6°53'38 'E. Initially, they undergo a manual extraction process. They are submerged in distilled water to remove any impurities on their surface. After that, the fibers are naturally air-dried for 15 days at room temperature (RT = 25 °C) to eliminate moisture. The WF fibers undergo treatment with a sodium bicarbonate solution (10% $NaHCO_3$), available at the Chemical engineering laboratory of the university of 20 August 1955-Skikda, for varying durations (0, 24, 48, 72, 120, and 168 h) at RT before the biocomposite fabrication.

PLA is the polymer employed as the matrix; its density is 1.26 g/cm^3 [64–67]. We used a Brabender Plastograph internal mixer to manufacture the biocomposite material, as shown in Fig. 1. We mixed 68.08 g of PLA (74% by weight) at 50 rpm and 180 °C for 2 min. We added 22 g of WF fibers cut between 2 and 3 cm (24% by weight) and 0.92 g of charcoal (1% by weight), derived from the combustion of the WF petiole, and left the mixture in the blender for 12 min to enhance the homogeneity of the material produced. Following the treatment, the mixture is then subjected to oven drying for 48 h at a temperature of 60 °C. Subsequently, biocomposite sheets are fabricated using a hot press set at 175 °C (hydraulic compression molding) for 10 min. The mold is then allowed to cool to 60 °C for 5 min. Fig. 1 illustrates the samples before and after the compression process. All experiments are conducted at the Department of Bio-composite Laboratory, Institute of Tropical Forestry and Forest Products, Universiti Putra Malaysia, 43, 400, UPM Serdang, Selangor, Malaysia. The terminology of the fibers and resulting biocomposites are outlined in Table 1.

2.2. Characterization methods

2.2.1. Scanning electron microscopy (SEM) analysis

The morphological characteristics of treated WF, untreated WF, and PLA/Bi-WF samples were analyzed using the topographic mode of the COXEM EM-30 Plus. An Au–Pd sputter coating was applied to the samples to guarantee electrical conductivity at the surface. The tests used an electrical acceleration voltage of 20 kV.

Table 1
Nomenclature of fibers and developed biocomposites (WF: *Washingtonia filifera*, PLA: polylactic acid, Bi: Biochar).

Treatment time (h)	WF fiber	PLA/Bi + WF
0	UWF	PLA/BiUWF
24	WF-24	PLA/BiWF-24
48	WF-48	PLA/BiWF-48
72	WF-72	PLA/BiWF-72
120	WF-120	PLA/BiWF-120
168	WF-168	PLA/BiWF-168

2.2.2. Fourier transform infrared (FTIR) analysis

This study evaluated the changes to the chemical functional groups of raw and processed WF fibers using PerkinElmer FTIR spectroscopy's attenuated total reflectance approach. The FTIR spectrum spans wavelengths from 4000 to 400 cm^{-1}.

2.2.3. Differential scanning calorimetry (DSC)-thermogravimetric analysis (TGA)

A DSC Q20 V24.11 Build 124 apparatus was used to analyze the thermal behavior of untreated and raw WF fibers. A 20 °C/min heating rate was applied for the DSC study, per ASTM D3418-82 standards [68]. Temperature ranges from 20 to 350 °C were utilized for the analysis. The mass of the samples used in this work is between 6 and 7 mg. TGA analysis of raw and treated WF fibers was performed on a TGA Q500 V20.13 Build 39 machine following ASTM E1131-03 [69]. The average sample mass used in this work is 10.5 mg under nitrogen with a flow rate of 50 mL/min. The WF fibers were heated at 10 °C/min for the thermal examination from 24 to 580 °C.

Fig. 1. Preparation of biocomposites polylactic acid (PLA)/biochar (Bi)-*Washingtonia filifera* (WF).

2.3. Mechanical characterization

Traction and flexion 3-point statistical tests were performed on the prepared biocomposites using a universal testing machine (Instron 5567) equipped with a force sensor of 30 KN to ascertain the impact of treatment time variation on the resistance of the resultant biocomposites hybrids. Using a molding machine, ASTM D638-14 [70] was followed in the fabrication of the test specimens for conducting tensile tests, and the 3-point bending test ASTM D790-17 standard [71]. For each formulation, the tests were repeated three times under natural climatic conditions, with a temperature of 25 °C and a relative humidity of around 44%.

2.3.1. Izod impact tests

For the Izod tests, an Instron CEAST 9050 equipped with a 0.5 J hammer was used. The strength and brittle ductility were assessed using the machine pendulum per the ASTM D256-10e1 standard [72]. Three specimens for each treatment duration were tested to guarantee the reliability and consistency of the results.

3. Results and discussion

3.1. Scanning electron microscopy (SEM) analysis

Fig. 2 illustrates the images obtained through SEM depict the transverse and longitudinal topographic surface of raw and sodium bicarbonate-treated (10% $NaHCO_3$) WF fibers for various treatment durations (24, 48, 72, 120, and 168 h). Since WF fibers are naturally exposed to climate changes, the fiber surface morphology is different and rough, as shown in Fig. 2a. Raw WF fibers exhibit surface impurities, such as natural oils and wax [73,74], without fiber fibrillation. Fig. 2 (a-f) shows that the raw WF fibers' surface is smoother and rougher than that of the WF fibers treated with sodium bicarbonate. The alkali action involves disrupting hydrogen bonds on the fiber surface [75–77], removing hemicelluloses and lignins, and increasing surface roughness. This increase in roughness promotes interfacial adhesion between fibers and the matrix, as a rougher surface facilitates the mechanical interlocking of fibers with the matrix. Consequently, as the processing time increases, fiber degradation also occurs [74], which may explain the

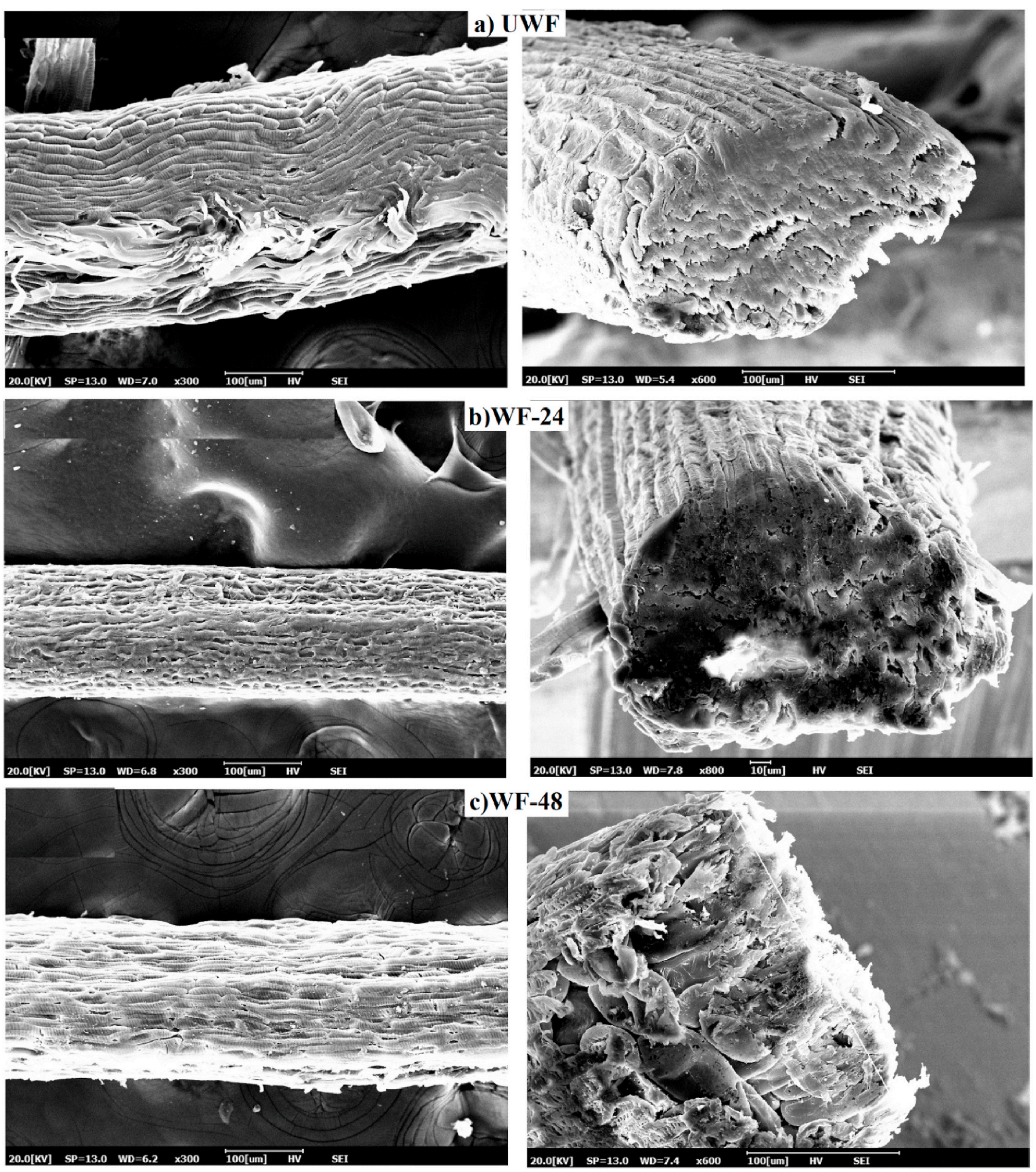

Fig. 2. Scanning electron microscopy (SEM) images of *Washingtonia filifera* (WF) fibers: a) untreated; treated with $NaHCO_3$ b) 24 h, c) 48 h, d) 72 h, e) 120 h, and f) 168 h.

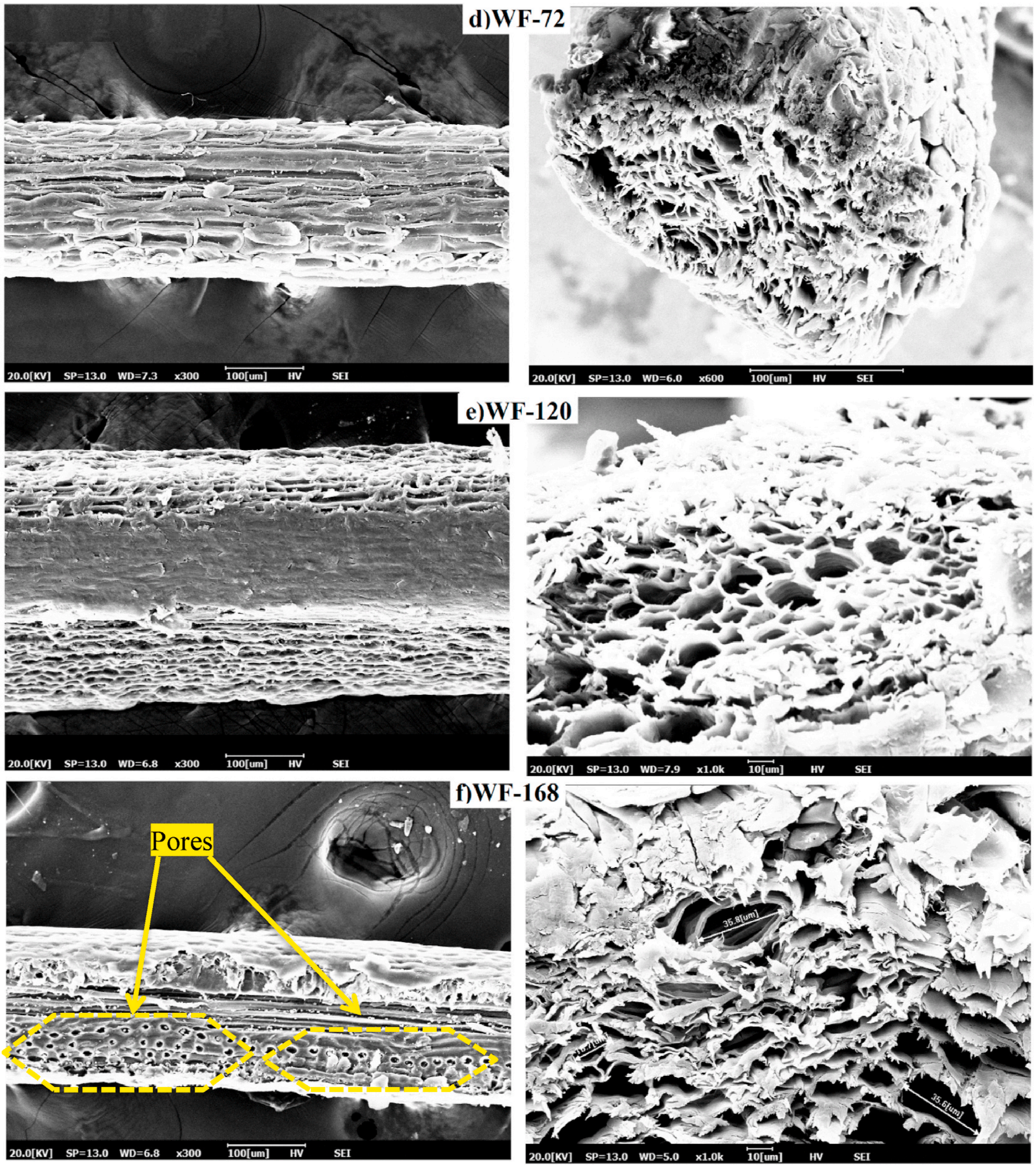

Fig. 2. (*continued*).

lower flexural and tensile strength obtained from the hybrid-developed PLA/BiWF-120 and PLA/BiWF-168. Fig. 3 shows the fractographs of the specimens used in the tensile strength tests. Micro-defects are recorded during the fabrication of hybrid biocomposites. These images show that the biocomposite contains pores and voids, the latter being due to fiber pull-out at the fractured site. It is evident that the PLA/BiUWF, PLA/BiWF-24, and PLA/BiWF-48 hybrid biocomposites exhibit poor interfacial adhesion compared to the PLA/BiWF-72 hybrid biocomposite, and the fiber pull-out in the micrographs confirms weak fiber/matrix adhesion (Fig. 3(a-c)). It is observed that fiber pull-out and fiber-matrix debonding are the essential causes of rupture in hybrid biocomposites subjected to tensile loading. The fractographs of the PLA/BiWF-72 and PLA/BiWF-120 hybrid biocomposites, shown in Fig. 3 (d-e), revealed a significant increase in surface bonding between the PLA matrix containing Bi and the treated WF fiber. Consequently, the surface's chemical treatment enhanced the compatibility of WF fibers with PLA-Bi.

3.2. Fourier transform infrared (FTIR) analysis

Fig. 4 show comparative FTIR spectra of treated and raw ($NaHCO_3$ solutions) WF fibers in the range of 500–4000 cm^{-1} with different treatment durations (24, 48, 72, 120, and 168 h). The presence of cellulose, hemicellulose, and lignin was demonstrated by the FTIR spectra [78]. Characteristic bands indicate the presence of hemicellulose in the fiber composition, typically observed around 1735 and 1245 cm^{-1} [79]. FTIR analysis enables us to precisely discern the chemical composition of WF fibers.

The spectrum of WF fiber reveals a prominent absorption band at approximately 3340 cm^{-1}, which is responsible for the stretching vibrations of hydroxyl groups (OH) present in cellulose molecules. Furthermore, two distinct bands are observed within the spectrum's 2800–2900 cm^{-1} region. These bands are primarily due to the stretching vibrations of the –CH (carbon-hydrogen) bonds characteristic of hemicellulose molecules [80,81]. The absorption band at 1735 cm^{-1} in the FTIR spectrum corresponds to the stretching vibrations associated with the carbonyl ester group C═O (carbon-oxygen double bond). The primary cause of this distinctive band is the hemicellulose content of the

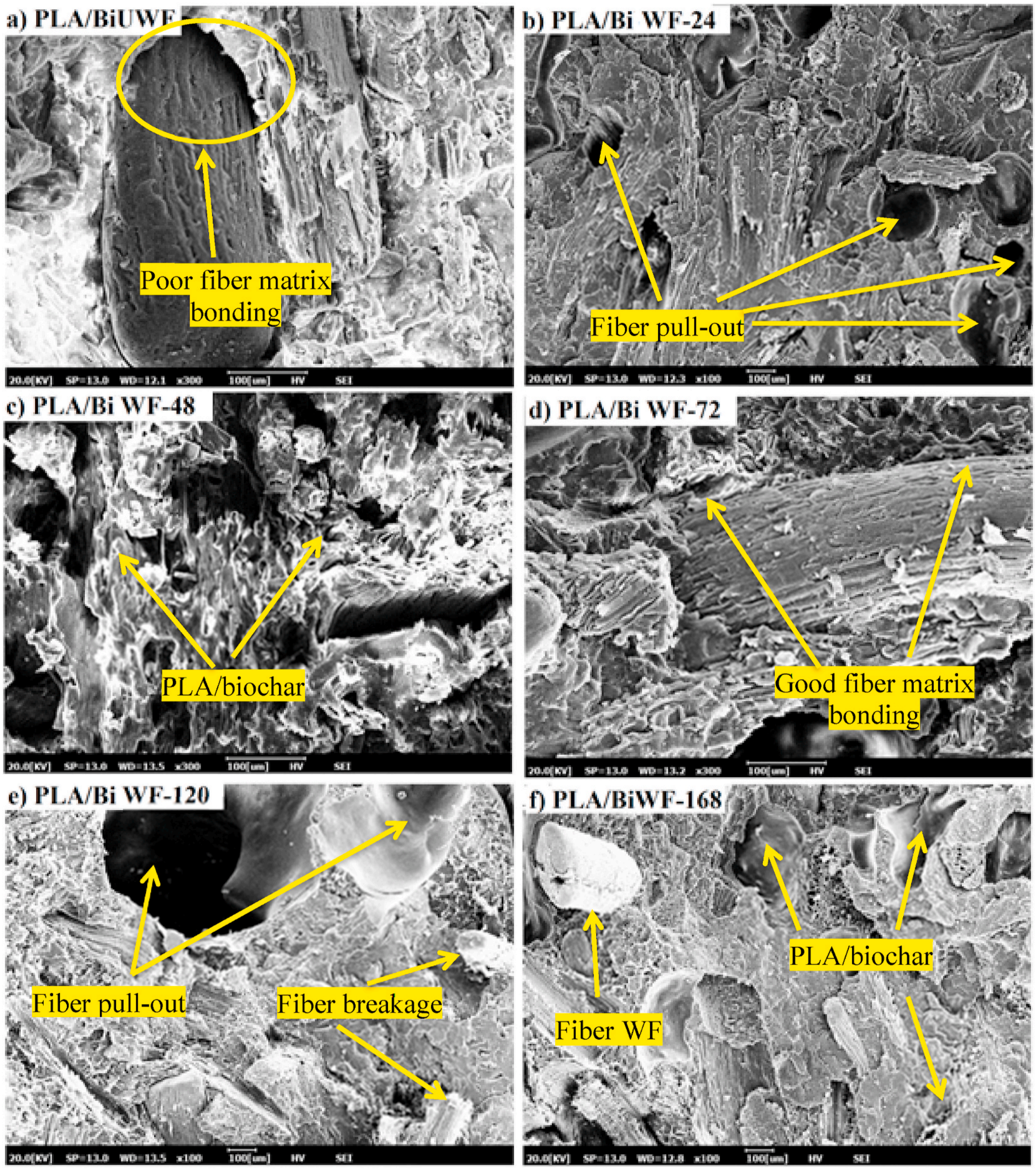

Fig. 3. Tensile test fractures of hybrid biocomposites.

sample. The band linked to C═C bond stretching, located at around 1645 cm^{-1}, suggests the existence of lignin. The band of the FTIR spectrum at 1030 cm^{-1} indicates the CO stretching vibration of the acetyl group, present in lignin and hemicellulose.

Additionally, the band at 1424 cm^{-1} further delineates the presence of cellulose within the sample and contributes to the comprehensive characterization of the WF fibers. Finally, in the FTIR spectrum, a prominent band is observed at 730 cm^{-1}, the vibration caused by the C–OH deformation [80,82]. For fibers treated with $NaHCO_3$ for 24, 48 and 72 h, the intensity of bands related to hemicellulose indicates a slight decrease. The significant decline in hemicellulose is more evident after 120 and 168 h of treatment. Other authors have already obtained similar results [47,73,79]: for sisal fibers and [83] for coir fiber treated with $NaHCO_3$. Similar changes in FTIR spectra were observed by Liu et al. [84] after treating natural grass fibers with an alkaline solution. These results indicate the partial elimination of hemicellulose and lignin from the WF fibers after exposure to sodium bicarbonate.

3.3. Thermogravimetric analysis (TGA)-Derivative analysis

TGA curves and the differential thermogravimetric (DTG) analysis illustrated in Fig. 5(a-b) showcase two-stage thermal degradation behavior observed in treated and untreated WF fibers. Fig. 5a offers valuable insights into the thermal evolution of both untreated and treated fibers, enabling a comprehensive discussion of the impact of WF fiber processing on their thermal stability. Additionally, it is evident that none of the examined WF fibers, regardless of the treatment duration, display any significant difference in thermal stability values. The thermal curves of the treated and untreated WF fibers indicate that cellulose decomposes at about 350 °C, whereas dehydration and lignin degradation happen between 243 and 375 °C [80,85]. Fiber sensitivity to moisture decreases progressively as treatment time increases [79]. The removal of lignin and hemicellulose during the sodium bicarbonate treatment causes the variance in weight loss between the treated and untreated WF fibers as the temperature increases. This observation aligns with the findings obtained from FTIR spectra analysis.

Fig. 5b shows a peak representing hemicellulose in the DTG curves of raw WF fibers and those treated with sodium bicarbonate for 24, 48 and

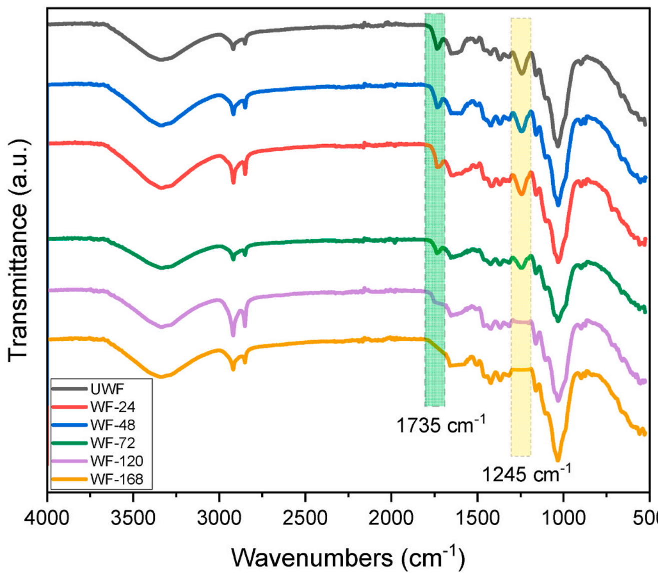

Fig. 4. Comparative Fourier transform infrared (FTIR) analysis of *Washingtonia filifera* (WF) fibers untreated and treated with $NaHCO_3$ for 24, 48, 72, 120 and 168 h.

72 h [86]. The intensity of this peak then decrease relative to the other fibers until it disappears completely, which is due to pyrolysis of the hemicellulose and is more evident in fibers treated for 168 h. The maximum degradation rates of treated WF fibers for the durations 24, 48, 72, 96, 120 and 168 h were found to be around 351, 338, 340, 340, 338 and 328 °C, respectively, while for the raw fiber, it was found to be at 351 °C. The leading cause of this deterioration is the cellulose's pyrolysis inside the fibers [87,88]. Table 2 shows the degradation results for raw and $NaHCO_3$-treated WF fibers over different periods. As per the findings, the thermal decomposition of cellulose and hemicellulose contributed significantly to char formation, resulting in a notable amount of char residue observed in WF fibers treated for 168 h (20%).

3.4. Differential scanning calorimetry (DSC) analysis

DSC thermograms obtained for WF fibers treated with sodium bicarbonate (10% $NaHCO_3$) for different treatment times (24, 48, 72, 120 and 168 h) have the same shape as untreated fibers (Fig. 6). The graphs show an endothermic peak centered at 161, 175, 175, 176 and 177 °C, respectively, for treated fibers, while for untreated fibers, it is centered at 174 °C, the temperature at which water evaporates. These small and large endothermic peaks on treated WF fibers indicate cellulose decomposition [89,90], while the exothermic peaks (250–310 °C) indicate complete hemicellulose decomposition [91]. The variation in enthalpy observed among different fibers corresponds to the energy required for evaporation. The rise in amorphous cellulose, characterized by its poor resistance to thermal stress ($T = 161.13$ °C), is the cause of the decrease in the breakdown temperature of fibers treated with NaOH for 24 h. The fiber treated with 10% $NaHCO_3$ for 24 h has an enthalpy of 126.7 J/g, but the raw WF fiber has a greater enthalpy ($\Delta H = 132.9$ J/g). On the other hand, the WF-24, WF-72 and WF-120 fibers have almost the same enthalpy values of 126.7, 124.6 and 126.8 J/g, respectively. Furthermore, the enthalpy of fibers treated for 48 h ($\Delta H = 149.9$ J/g) is higher than that of all the fibers studied. The enthalpy of WF-48 and WF-168 treated fibers may have increased because the cellulose chains were tighter. Still, the decrease in enthalpy values observed for the treated fibers WF-24, WF-72, and WF-120 could be attributed to the relaxation of the chain structure induced by $NaHCO_3$ treatment. This structural loosening likely facilitated the degradation process [89].

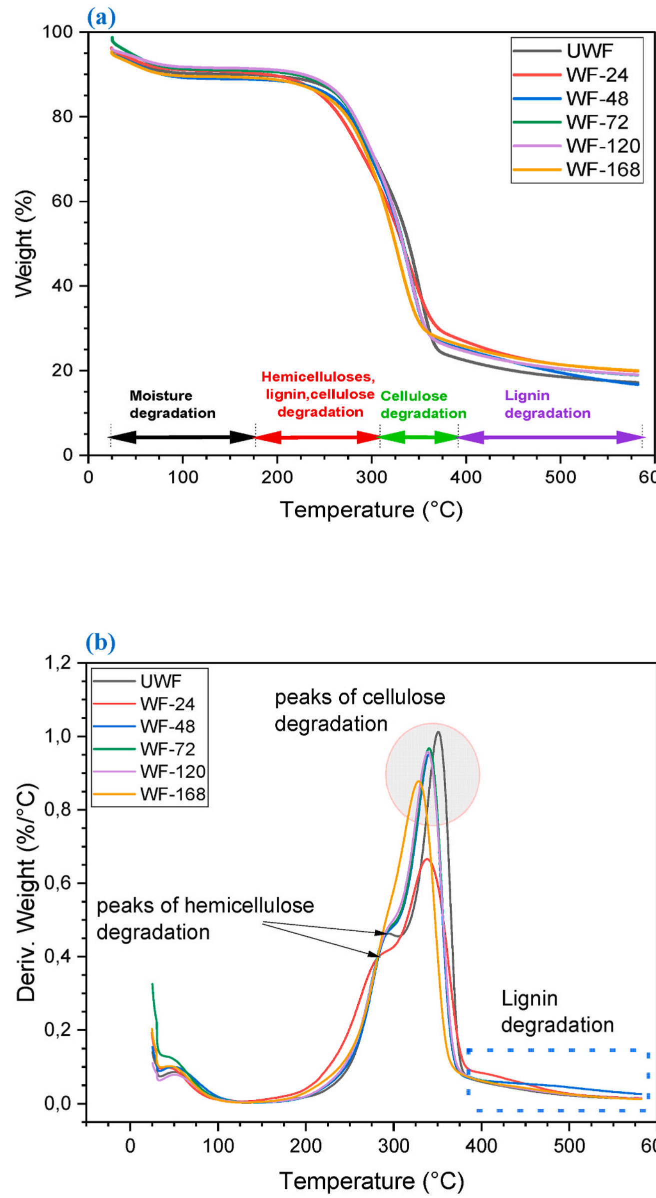

Fig. 5. Comparative thermogravimetric analysis (TGA) (a) and (b) DTG analysis of *Washingtonia filifera* (WF) fibers untreated and treated with $NaHCO_3$ for 24, 48, 72, 120, and 168 h.

3.5. Characterization of PLA/Bi-WF hybrid biocomposites using tensile and 3-point bending tests

Figs. 7a and 8a illustrate the effect of fiber treatment on the mechanical behavior in traction and flexion of PLA hybrid biocomposites based on Bi and WF fibers treated with sodium bicarbonate (10% $NaHCO_3$) for different treatment durations (24, 48, 72, 120 and 168 h). It is observed that all the developed hybrid composites have undergone brittle fracture. These results clearly show that treating WF fibers has improved flexural and tensile strength and elasticity modulus for the hybrid biocomposites compared to those reinforced with raw fibers. Similar results were found by Maio and Scaffaro [92] for green composites based on agricultural waste from chamaerops humilis. The tensile and flexural strength of the biocomposites exhibited a noticeable enhancement. This improvement in the strength of the developed hybrid biocomposite can be attributed to the treatment of fiber and biochar. Thanks to its unique structure and large surface area, biochar contributes to this improvement, which helps improve the interfacial adhesion

Table 2
Initial and maximum degradation temperature values for PLA/Bi-WF hybrid biocomposites from thermogravimetric analysis (TGA) thermograms (WF: *Washingtonia filifera*, PLA: polylactic acid, Bi: Biochar).

Material	First stage of degradation (°C)	Second stage of degradation (°C)	Temperature at 10% mass loss (°C)	Temperature at 50% mass loss (°C)	Coal residue at 580 °C (%)
UWF	25.40–99.86	251.96–372.77	169.63	339.28	17.19
WF-24	24.58–91.56	244.82–375.49	189.66	332.46	19.86
WF-48	25.34–99.22	247.17–366.39	179.58	332.62	16.70
WF-72	25.29–100.50	243.66–364.31	219.49	333.31	18.93
WF-120	25.37–96.35	243.60–368.46	227.92	332.915	18.99
WF-168	24.67–94.27	248.44–359.53	181.82	324.93	20.00

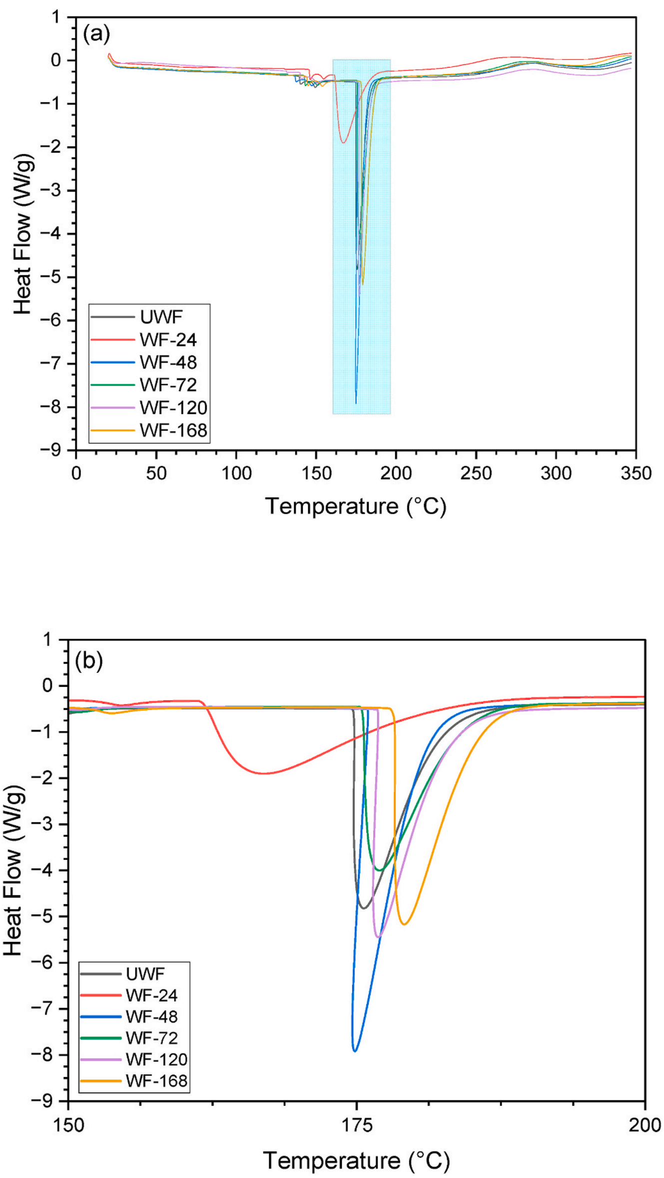

Fig. 6. Comparative differential scanning calorimetry (DSC) analysis of *Washingtonia filifera* (WF) fibers untreated and treated with $NaHCO_3$ for 24, 48, 72, 120, and 168 h. (a) Temperature 25–350 °C, and (b) zoom of the selected region between 150 and 200 °C.

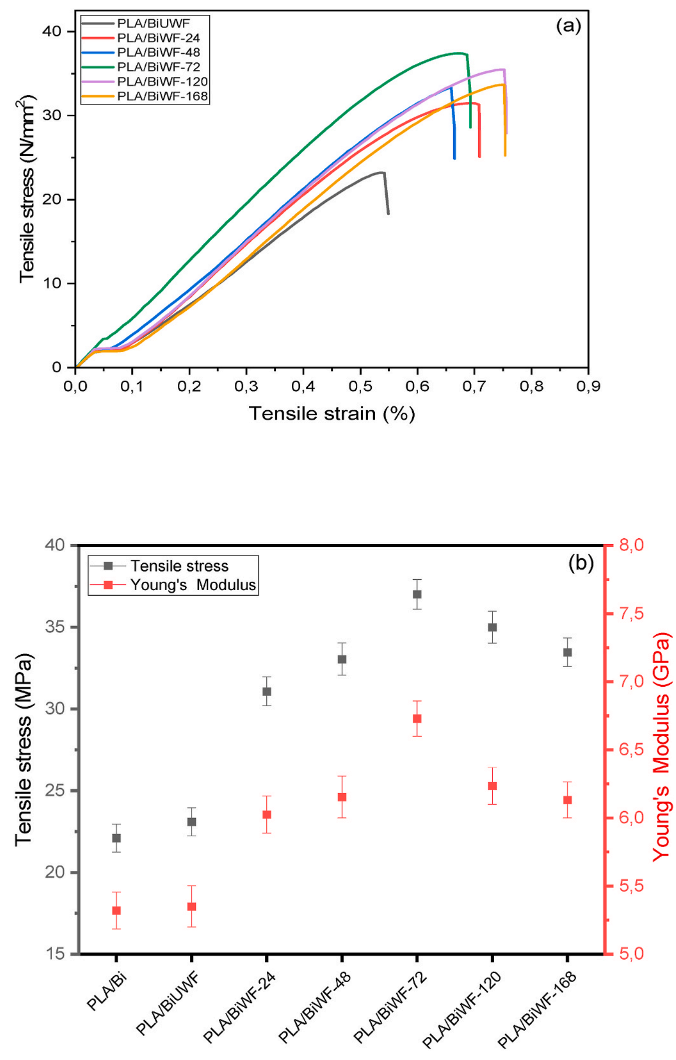

Fig. 7. Tensile properties of polylactic acid (PLA)/biochar (Bi)-*Washingtonia filifera* (WF) hybrid biocomposites. (a) Tensile stress, and (b) Young's modulus.

between biochar and PLA. The best performances are obtained with a 72 h treatment. As treatment duration increased, the tensile and flexural strengths of PLA/BiWF-72 hybrid biocomposites improved considerably. Treatment of the fibers for 72 h benefitted obtaining fibers with low hemicellulose residue content. These fibers are better able to interact with polymers than those that undergo complete hemicellulose removal. Residual hemicellulose promotes improved adhesion between fibers and polymers, thereby enhancing biocomposites' interaction and mechanical properties Importantly, this approach achieves an optimal balance between reducing hemicellulose residues and preserving the favorable characteristics of natural fibers [92]. Compared to the biocomposites manufactured, the biocomposites treated for 72 h had a slightly higher stress-strain curve. The PLA/BiUWF has an average strain at tensile rupture of 0.534% and at flexural rupture of 1.236%, while the PLA/BiWF-24, PLA/BiWF-48, PLA/BiWF-72, PLA/BiWF-120, and PLA/BiWF-168 have average strain at tensile rupture of (0.693, 0.659, 0.672, 0.752, and 0.752%) and at flexural rupture of (2.146, 2.838,

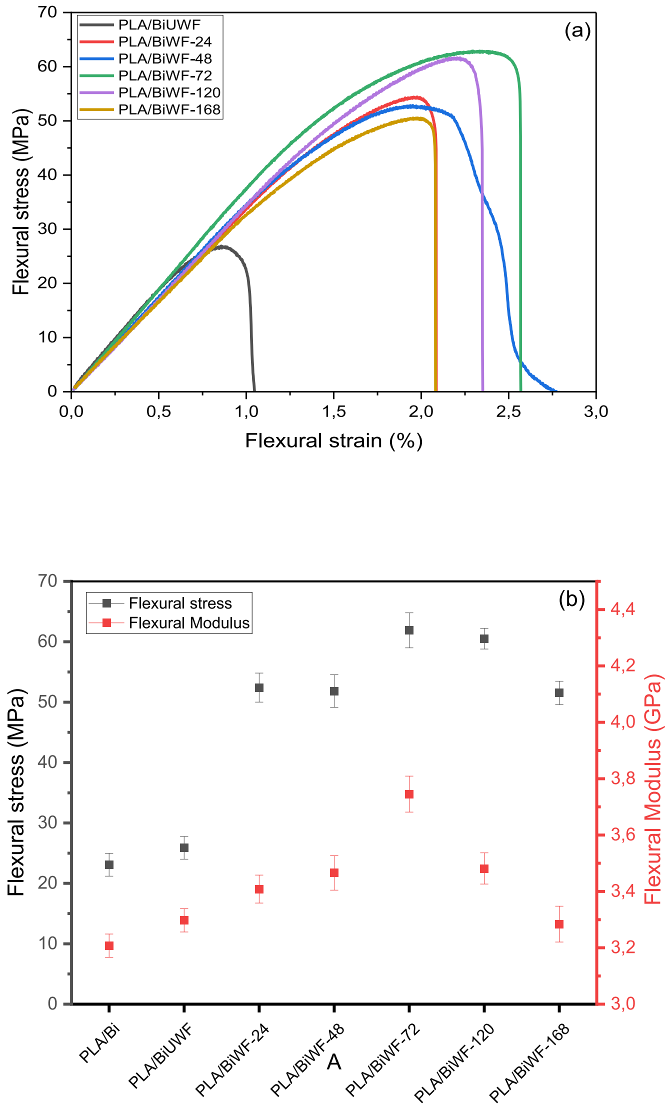

Fig. 8. Flexural properties of polylactic acid (PLA)/biochar (Bi)-*Washingtonia filifera* (WF) hybrid biocomposites. (a) Flexural stress, and (b) Flexural modulus.

3.380, 2.293, and 2.159%), respectively. The deformation was also minor, likely because of the weak cohesion between the matrix and fiber or poor mechanical interaction.

Figs. 7b and 8b highlight various mechanical characteristics, including the flexural modulus, elastic modulus, tensile, and flexural strength. These data were extracted from the tensile and flexural curves of the hybrid composites produced. Figs. 7b and 8b clearly show that the PLA/BiWF-72 hybrid biocomposites have higher tensile strength (37.41 ± 0.91 MPa) and flexural strength (61.89 ± 2.89 MPa) compared to the other studied biocomposites. A decreased flexural strength (51.53 ± 1.92 MPa) and tensile strength (33.46 ± 0.86 MPa) were observed for the 168-h processing time. The findings show that when processing time increases, the elastic modulus increases. In particular, the PLA/BiWF-72 biocomposite exhibited an elasticity modulus of 6.729 GPa and a flexural modulus of 3.745 GPa. An improvement of 20.49% and 11.96% in tensile and flexural modulus were observed, respectively, compared with PLA/BiUWF biocomposite. In addition, the PLA/BiWF-72 hybrid exhibited an increase of 37.57% in tensile strength and 58.18% in flexural strength compared to the PLA/BiUWF biocomposite. In contrast, 26.12 and 75.23% decreases are recorded for the same mechanical characteristics of PLA/BiWF-168 biocomposites compared to PLA/BiWF-72. Good adhesion (WF/PLA) can explain the increased strength of the PLA/BiWF-72 hybrid, which translates into stress transfer between the PLA and the treated WF fibers. In addition, prolonged treatment periods (over 72 h) were also associated with a decrease in tensile strength due to the deterioration of the fibers caused by an extended treatment time.

In this study, the flexural strength of the PLA/BiWF-72 hybrid, which is of the order of (61.89 ± 2.89 MPa), is higher than that obtained by Makhlouf et al. [80] (19.72 ± 2.52 MPa) for the HDPE/10% flax fiber composite. On the other hand, the tensile modulus (5.35 ± 0.15 GPa) and flexural modulus (3.29 ± 0.04 GPa) for the PLA/BiWF-24 hybrid biocomposite are higher than those found by Dos Santos et al. [79] for epoxy-based biocomposites reinforced with sisal fibers treated with bicarbonate for 24 h, with respective stresses of (σ_t = 4.70 ± 0.01 GPa) and (E_f = 3.13 ± 0.16 GPa). The tensile strength obtained (37.41 MPa) for the PLA/BiWF-72 biocomposite is superior to those found by Sarmin et al. [93] (24.04 MPa) for the bio-epoxy composite based on palm fibers and by Faris et al. [94] for polypropylene biocomposites reinforced with DPFs, but lower than that obtained by Chaitanya and Singh [73] for PLA biocomposite based on treated sisal fibers at 72 h (56.01 MPa).

3.6. Izod impact test of PLA/Bi-WF biocomposites

The Izod impact test is a procedure used to assess impact resistance and measure a material's ability to absorb and dissipate impact forces and loads. It is another test for evaluating the structural properties of materials [95,96]. Fig. 9 represents the variation in impact resistance of PLA hybrid biocomposites reinforced with Bi and WF fibers treated with sodium bicarbonate (10% $NaHCO_3$) for different treatment times (0, 24, 48, 72, 120, and 168 h). The impact resistance of the hybrid composites produced is significantly impacted by the sodium bicarbonate treatments applied to the WF fibers, as seen by the results of the Izod impact tests. We found that the best impact resistance of the hybrid biocomposite was that of the PLA/BiWF-24 biocomposite (3.453 ± 0.197 kJ/m^2). The impact resistance gradually decreases for biocomposites reinforced with WF fibers treated beyond 24 h. The impact resistance of the PLA/BiWF-72 (2.94 ± 0.129 kJ/m^2), PLA/BiWF-120 (2.82 ± 0.152 kJ/m^2), and PLA/BiWF-168 (2.805 ± 0.181 kJ/m^2) hybrid composites was found to decrease, as shown by the results of the Izod impact tests.

Some authors have already observed reduced biocomposites based on plant fibers [97,98]. The decrease in impact resistance of PLA/BiWF-72, PLA/BiWF-120, and PLA/BiWF-168 biocomposites is due

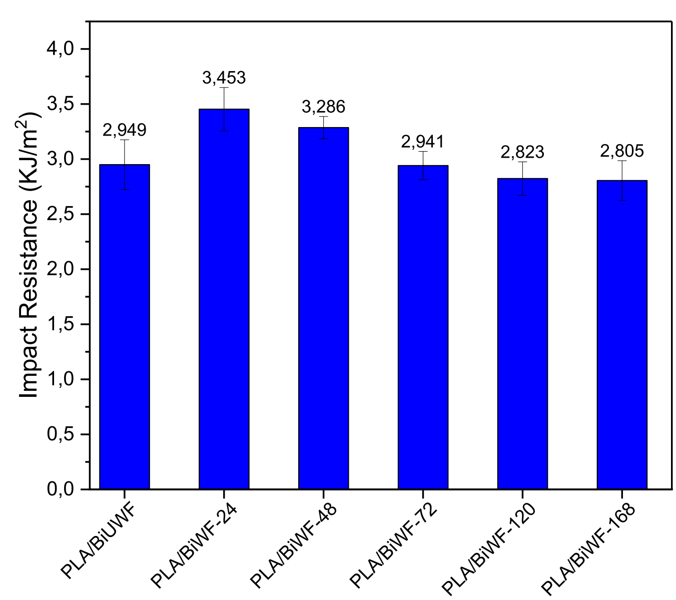

Fig. 9. Izod impact resistance of hybrid biocomposite polylactic acid (PLA)/ biochar (Bi)- *Washingtonia filifera* (WF) products.

to the improved cohesion between the PLA and treated WF fibers, resulting in a higher number of fiber fractures during impact testing. Additionally, fiber pull-out dissipates more energy than fiber fractures [99–101], leading to a lower impact resistance of the developed hybrid biocomposites [73]. The impact resistances of the two hybrid biocomposites, PLA/BiWF-24 and PLA/BiWF-48 increased by 17% and 11.42%, respectively, compared to the biocomposite PLA/BiWF. However, the biocomposite PLA/BiWF-168 exhibited the lowest impact resistance (2.805 ± 0.181 kJ/m^2) compared to the other developed biocomposites. The impact resistances of the PLA/BiWF-24 (3.453 kJ/m^2) and PLA/BiWF-48 (3.286 kJ/m^2) biocomposites are higher than those achieved by Tokoro et al. [100] for the PLA biocomposite reinforced with treated bamboo fibers (1.51 kJ/m^2) and by Sarmin et al. [93] for the bio-epoxy composite based on DPFs (2.39 J/m^2). On the other side, the impact resistance of the PLA/BiWF-24 hybrid biocomposite (3.453 kJ/m^2) is lower than that reported by Satapathy and Kothapalli [102] for recycled HDPE composite based on banana fibers (11.79 kJ/m^2) and by Koffi et al. [98] for the HDPE biocomposite reinforced with birch fibers (4.43 kJ/m^2). In addition, the impact resistances of the hybrid biocomposites produced are lower than those found by Chen et al. [103] (19 kJ/m^2) for the HDPE composite based on poplar wood fibers by Shang et al. [104] (3.58 kJ/m^2) for the HDPE composite reinforced with yellow pine fibers.

4. Summary and conclusions

We studied the thermal behavior of *Washingtonia filifera* (WF) fibers treated with sodium bicarbonate (10% $NaHCO_3$) for varying treatment durations (24, 48, 72, 120 and 168 h). Several results were obtained on the mechanical characterization and influence of different treatment times on the fiber surface of a polylactic acid (PLA)-based hybrid biocomposite material incorporating biochar (Bi) and WF fibers. This study led to the following conclusions.

- Treating WF fibers with sodium bicarbonate results in the incomplete elimination of lignin and hemicellulose, as confirmed by Fourier transform infrared (FTIR) and thermogravimetric analysis (TGA). The significant decrease in hemicellulose is most evident after 120 and 168 h of treatment.
- TGA results showed that a considerable quantity of Bi residue (20% coal residue) was found in the WF fiber samples treated for 168 h because of the thermal decomposition of hemicellulose and cellulose.
- Amorphous cellulose, characterized by its low resistance to thermal stress, is responsible for the decrease in the decomposition temperature of fibers treated with $NaHCO_3$ over 24 h ($T = 161.13$ °C).
- Scanning electron microscopy (SEM) observations show that chemical treatments improve fiber adhesion to the matrix and remove impurities from the fiber surface.
- Based on the findings from the static 3-point tensile and flexural tests performed on the manufactured hybrid biocomposite materials, integrating treated WF fibers and Bi into a PLA matrix enhances mechanical proprieties, including flexural and tensile strength, flexural modulus, and elasticity modulus. Remarkably, the most notable enhancements in mechanical characteristics are observed for the PLA/BiWF-72 biocomposite. When processing time increased, the modulus of elasticity rose, and the PLA/BiWF-72 biocomposite exhibited tensile and flexural moduli of 6.729 and 3.745 GPa, respectively. Deformation was also minor, probably due to poor mechanical interaction or weaker adhesion between WF fibers and PLA.
- The debonding of matrix fiber and the pull-out of fibers are the leading causes of failure in hybrid biocomposites developed under tensile loading.
- The results obtained from the Izod impact tests demonstrate the notable influence of sodium bicarbonate treatments on the WF fiber on the impact resistance of the hybrid produced. PLA/BiWF-24 biocomposite exhibited the highest impact resistance among the composites produced (3.453 ± 0.197 kJ/m^2).
- The inclusion of Bi and the 72 h treatment of WF fibers exhibited their capability to enhance the fibers' mechanical properties. This process's PLA/BiWF72 biocomposite showed the best mechanical properties increase. The enhancement can be partially attributed to the reduced hemicellulose residue content in the fibers obtained after the 72 h treatment. These fibers, with a residual amount of hemicellulose, are more conducive to interactions between fibers and polymers than those that have undergone complete hemicellulose removal (the 120 and 168 h treatments). This promotes better adhesion and compatibility between the biopolymer matrix and fibers, leading to significant improvements in the biocomposites' mechanical characteristic.
- The enhanced strength of the developed hybrid composite is attributed to the processing of fibers and Bi. This is primarily due to their unique structure and large surface area, facilitating improved interfacial adhesion between the Bi and the matrix.

The hybrid biocomposites we successfully developed from plant materials such as WF fibers, Bi, and PLA polymers are biodegradable, non-toxic, and environmentally friendly. These biocomposites can be used in various structural and non-structural commercial products, such as streetcar and train interiors, biomedical equipment and materials, sports equipment, electronic components, food packaging.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jmrt.2024.06.033.

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